

# Appendix H

## Detached solidification of InSb on earth

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### Abstract

Detached solidification of lightly gallium-doped indium antimonide was achieved in the laboratory only when the ampoule was coated with hexagonal boron nitride and when the material appeared to be oxide-free. A furnace was constructed with the temperature increasing with height in order to minimize buoyancy-driven convection, so as to maximize transport of segregated dissolved gases into the gap between the growing solid and the ampoule wall. There appeared to be no difference in results with freezing rates of 5 mm/h and 10 mm/h. Best results were obtained when the ampoule was backfilled with 20 kPa of Ar-10% $H_2$  prior to sealing. The detached portions were depressed by several  $\mu m$  from adjacent attached regions, were rough, and sometimes had microfacets and steps.

*PACS:* 81.05.Ea; 81.10.-h; 81.10.Mx.

*Keywords:* A1. Directional solidification; A.2. Bridgman technique; B1. Indium antimonide; B2. Semiconducting III-V materials

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### 1. Introduction

In detached solidification the solid grows without contact with the ampoule wall, i.e. there is a separation between some portion of the ingot and the wall. As reviewed in [1], detached ingots were first observed from several experiments performed on Skylab in 1974, and subsequently in many experiments carried out in orbiting spacecraft (microgravity) on a wide variety of materials. Although reproducibility was poor, detachment occurred more often with ampoules that did not stick to the solid, ampoules that were sealed in a vacuum, and with a low freezing rate [1]. Detachment was often observed to result in dramatically improved crystallographic perfection, including orders of magnitude fewer dislocations and no nucleation of grains and twins at the ampoule wall. Its cause remained a mystery until explained by the Moving Meniscus Model in the 1990's [2-10]. Paper [10] is believed to provide the best physical insight into the phenomenon, as well as the influence of gravity. Basically, there is a gas-filled gap between the growing solid and the ampoule wall. A meniscus connects the edge of the solid to the ampoule wall, with the remainder of the melt contacting the wall. The gas pressure in the gap equals that above the melt if the two ends of the ampoule are open and connected, and if attached growth has not occurred below the gap. On the other hand, if the seed end of the ampoule is closed, then the gas dissolves into the melt, is segregated out by the growing solid, and is liberated into the gap. In this case, the gas pressure in the gap will be different from that

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at the top end of the melt. Steady-state detachment is predicted to be favored by a high contact angle of the melt on the ampoule wall (i.e., "non wetting"), a low vapor-melt surface tension, and a high growth angle. When the seed end of the ampoule is sealed, additional factors favoring detachment are a high pressure of residual gas above the melt, a high solubility of this gas in the melt, low solubility of dissolved gas in the solid, a low freezing rate, and a large diffusion coefficient of the dissolved gas in the melt. Roughening of the ampoule wall can also be helpful [e.g., 2].

Several mechanisms have been proposed for the initiation of detachment. In one, a seed is used with a smaller diameter than the ampoule. The solidification begins detached provided the seed has not been completely melted back. Without such seeding, as solidification proceeds the stress due to differential thermal contraction between the cooling crystal and the ampoule increases until it is sufficient to break the adhesive bond between the two (for a non-ductile crystal with greater thermal expansion coefficient than the ampoule material). Thus ampoule surfaces that adhere poorly to the crystal should favor initiation of detachment. Such surfaces also are generally poorly wetted by the melt, which, as indicated above, favors continuation of detached growth. A third proposed mechanism is the formation of a gas bubble or tube at the ampoule wall that subsequently propagates around the periphery of the growing solid. Experiments with freezing of ice did not support this last mechanism [11].

It is highly likely that detached solidification has often occurred in terrestrial experiments without being recognized. To understand the influence of gravity, let us consider vertical Bridgman growth. The hydrostatic pressure in the melt increases the gas pressure required in the gap in order to maintain the meniscus, thereby increasing the required flux of gas into the gap. Gravity also causes buoyancy-driven convection. If this convection is vigorous, it causes dissolved gas rejected by the freezing interface to be mixed into the melt and subsequently liberated back into the top surface of the melt rather than into the gap. Continued detached growth is impossible. On the other hand, gentle convection that is radially outward directed at the freezing interface can increase the flux of segregated gas into the gap. For most materials, such gentle outward-directed flow is expected with a slightly convex freezing interface and with the temperature increasing with height throughout the melt. A convex interface shape tends to occur when the freezing interface is located at a point in the furnace where the temperature exceeds the material's melting point [e.g., 12].

Recently, with the recognition of detached solidification as an interesting and useful phenomenon, there have been several reports of detachment in vertical Bridgman or gradient-freezing experiments on the earth. In one experiment using a mirror furnace, detachment of Ga-doped germanium was observed *in-situ* and videotaped [13-17]. One could readily see the difference between the reflections of the melt in contact with the ampoule wall, the detached solid, and the meniscus as a band between them. The freezing interface was slightly convex, as predicted above. Similar results were obtained with Ga-doped Ge using closed-bottom pyrolytic boron nitride (pBN) containers [18,19]. The crystals grew attached to the wall if the pBN tube was open at both ends. Si-doped Ge has also been grown detached in pBN crucibles closed at the bottom [20-22]. HgZnTe grew detached if a carbon getter was included in the fused silica ampoule [23,24]. InSb in fused silica grew detached both in Mir and on earth, although the surface features were entirely different [25,26].

Duffar and coworkers showed how to deliberately pressurize the gap in order to maintain the required meniscus shape in GaSb growth [27,28]. The ampoule was sealed at the bottom and a seed was used with a smaller diameter than the ampoule. In [27], the pressure in the gap was

controlled via a gas tube connected into the side of the ampoule. Problems were encountered with traces of oxygen in the gas system. To avoid this, a completely sealed ampoule was used in [28], with a large inert gas volume below the seed in a third furnace. As solidification progressed, the temperature in the gas above the melt decreased and the hydrostatic pressure at the meniscus decreased, causing a net gas flow from the bottom into the gap. Excess gas pressure in the gap was relieved by emission of gas upward from the meniscus, so that maintenance of the required pressure was automatic.

The goal of the present research was to determine the influence of the operating conditions on the tendency to achieve detached solidification of InSb in our laboratory.

## 2. Experimental methods

Based on the above literature, it seemed that detachment would be favored by a high contact angle for the melt on the ampoule wall, an ampoule sealed at the bottom, a reasonable pressure of inert gas in the ampoule, a low freezing rate and a melt temperature increasing with height. Boron nitride has been established as producing the highest contact angles for semiconductor melts, including InSb [29-34]. Although a pBN crucible could have been used inside a silica ampoule, because of the unusual thermal properties of pBN this is thought to lead to difficulties in controlling the shape of the freezing interface. Additionally, it is not possible to see the ingot prior to removal. Previously, this laboratory had used borazine to coat the interior of ampoules for growth of CdTe [35]. However, borazine has become very difficult to obtain. Consequently, a new method was developed to coat the interior of silica growth ampoules with a film of transparent, very smooth and tightly-adhering hexagonal boron nitride [36,37]. This method was used to prepare some of the ampoules used here. Other ampoules were fused silica alone whose surface had been cleaned. All were 9-mm inside diameter and ~150-mm long with conical bottoms.

The experimental methods are described in detail in [37]. The polycrystalline InSb starting material contained approximately 1 ppm C and O, and 0.1% Ga.<sup>1</sup> Pieces were cleaned in hydrochloric acid and rinsed in water and methanol in order to remove native oxide. About 16 g of this material was loaded into a silica tube, which was heated to remove chemisorbed water, and alternately evacuated and filled with forming gas (Ar + 10% H<sub>2</sub>). The ampoule was sealed with 10, 20, 40 or 80 kPa of forming gas inside.

A specially constructed vertical Bridgman-Stockbarger furnace was used for the experiments. Nichrome heating wire was wound around a 2-cm ID silica tube, with the spacing between windings decreasing with height. Two copper tubes were inserted into the silica tube and separated by a 20-mm long fused silica tube, which served as an insulating zone to yield a longer section with a nearly constant temperature gradient. A 3-cm thick layer of ceramic fabric was wrapped around the fused silica. For temperature control, a K-type thermocouple was inserted between the fused silica tube and the top copper tube. Figure 1 shows the resulting temperature profile (in an empty ampoule) for two different temperature settings, and the position of the ampoule part way through an experiment. The ampoule was held entirely in the upper hot zone for 15 h, and then slowly lowered until it was entirely within the cooler bottom zone.

After an experiment, the ampoule was dissolved in 49% hydrofluoric acid. The ingot was rinsed with methanol. The resulting ingots were characterized by visual observation, optical

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<sup>1</sup> Glow discharge mass spectrometry performed 18 March 2003 by A. Mykytiuk and B. Methven at the Institute for National Measurement Standards, National Research Council of Canada, Ottawa.

microscopy, Energy Dispersive X-ray Spectrometry (EDS), Scanning Electron Microscopy (SEM), and stylus profilometry.

### **3. Experimental results**

The solidified ingots ranged from 4 to 6-cm in length and were 9-mm in diameter with conical bottoms. Table 1 summarizes the experiments performed and the results. Total detachment” signifies that the detached area went entirely around the circumference of the ingot, while “partial detachment” indicates that the detached area went only part way around the ingot or occurred on a small area. Most of the surfaces of the ingots were smooth with some gas bubble indentations on them, proving that the solid grew attached to the ampoule wall and replicated its surface except for the presence of gas bubbles on the wall. Detachment was indicated by a rough and dull surface, often with tiny facets, and without bubble indentations. Some ingots were partially or totally covered with a brown or black coating, presumably oxide. A uniform brown or black surface happened only when the ampoule leaked during experiments, such as with ingots 1 and 13. Sometimes a rough black surface occurred only on the top section of an ingot. Areas with such a brown or black coating were never detached. On some ingots that had a totally detached section near the top of the ingots, a short section above this detached area was shiny and smooth. Ingots 4, 5, 6, 15 and 19 clearly had detached portions, with clear boundaries between detached and attached areas. Several other ingots appear to have had detached areas, but without clear boundaries.

Table 1. Summary of experimental conditions and results

Expt. Number	Experimental Conditions				Results (Surface features of ingots)
	Pressure (kPa)	Ampoule Surface	Translation Rate	Furnace Temp. Set	
1	20	uncoated	10 mm/h	550 °C	The whole surface brown and rough (presumably oxide)
2	20	uncoated	10 mm/h	550 °C	2 h soak time. Surface not very shiny but quite smooth. About 8-mm black section on top.
3	20	uncoated	10 mm/h	550 °C	Some shiny and some dull areas. About 7-mm black section on top.
4	20	BN coated	10 mm/h	550 °C	Shiny and dull areas with sharp boundaries between them. Many gas bubble indentations on shiny areas (indicating attachment). Dull areas detached. About 20% of surface totally detached.
5	20	BN coated	8 mm/h	550 °C	Shiny and dull areas with sharp boundaries between them. Many gas bubble indentations on shiny areas (indicating attachment). Dull areas detached. About 20% of surface totally detached.
6	20	BN coated	10 mm/h	550 °C	Shiny and dull areas with sharp boundaries between them. A few gas bubble indentations on shiny areas (attached). Dull areas detached. Large cavities normal to growth direction. About 20% totally detached and partially detached nearly everywhere else.
7	-0	BN coated	10 mm/h	550 °C	Entirely black. Ampoule broke during experiment.
8	10	BN coated	10 mm/h	550 °C	Entire surface very rough, except for about a 3-mm shiny area on the top.
9	10	BN coated	10 mm/h	550 °C	Entire surface very rough.
10	50	uncoated	10 mm/h	550 °C	Entire surface shiny and smooth.
11	50	BN coated	10 mm/h	550 °C	Entire surface shiny and smooth, except for about 14-mm of black, rough area on the top. A few gas bubbles. Tiny facets on parts of the surface, indicating detachment.
12	80	BN coated	10 mm/h	550 °C	The whole surface shiny and smooth, except for a black, rough area on the top. A few gas bubbles.
13	10	BN coated	5 mm/h	550 °C	Entirely black. Ampoule broke during experiment.
14	10	BN coated	5 mm/h	550 °C	Entirely black. Ampoule broke during experiment.

15	20	BN coated	5 mm/h	550 °C	Shiny and dull areas with sharp boundaries between them. Large cavities normal to the growth direction. About 15% totally detached and about 20-mm partially detached below the totally detached section.
16	40	BN coated	5 mm/h	550 °C	The whole surface shiny and smooth, except for a black, rough area on the top.
17	40	BN coated	5 mm/h	550 °C	Shiny and rough areas. Totally shiny on the top. Boundaries between shiny and dull areas not as sharp as for Ingots 4, 5, 6 and 15.
18	10	BN coated	5 mm/h	550 °C	The whole surface shiny and smooth, except for a black, rough area on the top. Tiny facets on parts of the surface, indicating detachment.
19	20	BN coated	5 mm/h	580 °C	Shiny and dull areas with sharp boundaries between them. Large cavities normal to the growth direction. About 7% totally detached.
20	10	BN coated	5 mm/h	580 °C	Most of the surface shiny with gas bubbles. Partial detachment.
21	20	uncoated	5 mm/h	580 °C	Most of the surface shiny. Partial detachment.
22	20	BN coated	10 mm/h	580 °C	Large portion dull and rough without sharp boundary between shiny and dull. Tiny facets on parts of surface.

Fig. 2 illustrates an ingot exhibiting attached, partially detached, and fully detached regions. The boundary between attached and detached regions is shown at higher magnifications in Figs. 3 and 4. Fig. 5 illustrates an ingot grown attached with the exception of many small bubble/voids and a few large cavities. Fig. 6 shows a pair of photos of the boundary between attached and detached regions, with one focused on the attached plateau and the other on the detached valley. Fig. 7 is another optical micrograph showing microfacets and growth steps on one detached region. Such features can occur only in a detached region, and were sometimes seen when detachment was not otherwise apparent. Fig. 8 is a scanning electron micrograph of a boundary between attached and detached regions. Note the small steps on the rough detached surface.

Figs. 9 and 10 show stylus profilometer readings of transitions between attached and detached regions. There was approximately an 8- $\mu\text{m}$  difference in elevation between the detached and attached surfaces.

To verify that the rough surface of detached regions was not caused by an impurity such as oxygen, EDS was used to examine both dull and rough surfaces on clean ingots. No gallium or other foreign element was found either on rough or smooth surfaces, while the black or brown oxide on some ingots was highly enriched in Ga (as expected from the much higher free energy of formation of  $\text{Ga}_2\text{O}_3$  than  $\text{In}_2\text{O}_3$ ).

Efforts were made to find the most favorable conditions for detachment. Thus, different experiment conditions were used, as shown in Table 1. Two kinds of ampoule surface were used for the experiments. One was cleaned fused silica. The other was boron nitride coated fused silica. With a BN coating, the boundary between detached and attached sections was usually sharp. On the rare occasion when attachment occurred with an uncoated ampoule, it was only partial and the boundary between rough and smooth sections was not clear.

Prior experience and theory show that detachment should be favored by a lower freezing rate. Most ingots were grown either at 10 mm/h or at 5 mm/h. Although somewhat different results were observed at the two rates, detachment was not enhanced at the lower rate. Total detachment was observed near the top of two ingots at both translation rates. Ingot 6, grown at 10 mm/h, had a higher proportion of dull and rough surface than did Ingot 15, which was grown at 5 mm/h. However, they had almost same length of total detachment near the tops of the ingots.

Fig. 1 shows the temperature profile at two different furnace temperature settings, 550 and 580°C. At first, experiments were conducted with a temperature setting of 550°C. Detachment was observed on ingots grown under different conditions. Subsequently, the solid-melt interface was moved to a lower position in the furnace by increasing the temperature setting to 580°C. This should have made the freezing interface less convex and decreased the outward convection of melt near the interface. The ingots grown at different temperature settings showed different results. The detached portion was clearly distinguished from the attached section on Ingots 6 and 15, which were grown at a temperature setting of 550°C. Detachment also happened on Ingot 19, which was grown at a temperature setting of 580°C. Total detachment was found and the boundary between detached and attached surfaces was not as sharp as for Ingots 6 and 15.

The pressure of forming gas remaining in the ampoule before sealing ranged from 10 to 80 kPa. All of the ingots with total detachment were grown with 20 kPa. One ingot grown with 10 kPa had a surprisingly rough surface on the whole surface, as shown in Figure 4-21. It is believed that the solid had not melted completely before growth because later experiments did not show a similar result. Detachment was not conspicuous for the ingots grown at or above 40 kPa, although partially detached sections or tiny facets were found on the surfaces of some of

them.

#### 4. Conclusions

The two most important factors in achieving detachment here was to coat the interior of the growth ampoules with boron nitride and to avoid oxidation, which we found difficult to do. Best results were obtained with 20 kPa of Ar-10% $H_2$  in the ampoules prior to sealing. Freezing rates of 5 and 10 mm/h made little difference. The non-oxidized attached regions were smooth and shiny, except for small gas bubbles or voids. The detached regions were a few  $\mu m$  lower than the attached regions and were rough, sometimes with microfacets and steps. The ridges often seen in microgravity experiments were never observed here. Neither were striations. Our laboratory has rather a high vibration level, and so some of the variations in topography of the detached regions could have arisen from that. The influence of vibration (or g-jitter in space) may be critical for detached solidification, but has yet to be investigated. In no case was an ingot fully detached over its entire surface. The approach pioneered by Duffar and coworkers [28] seems more profitable for exploitation as a commercial method.

#### Acknowledgements

This research was supported by NASA Grant NAG 8-1482. We are grateful to William Micklethwaite of Firebird Semiconductors for his advice, interest and encouragement. We greatly appreciate our stimulating discussions and emails with Thierry Duffar and with Frank Szofran and co-workers.

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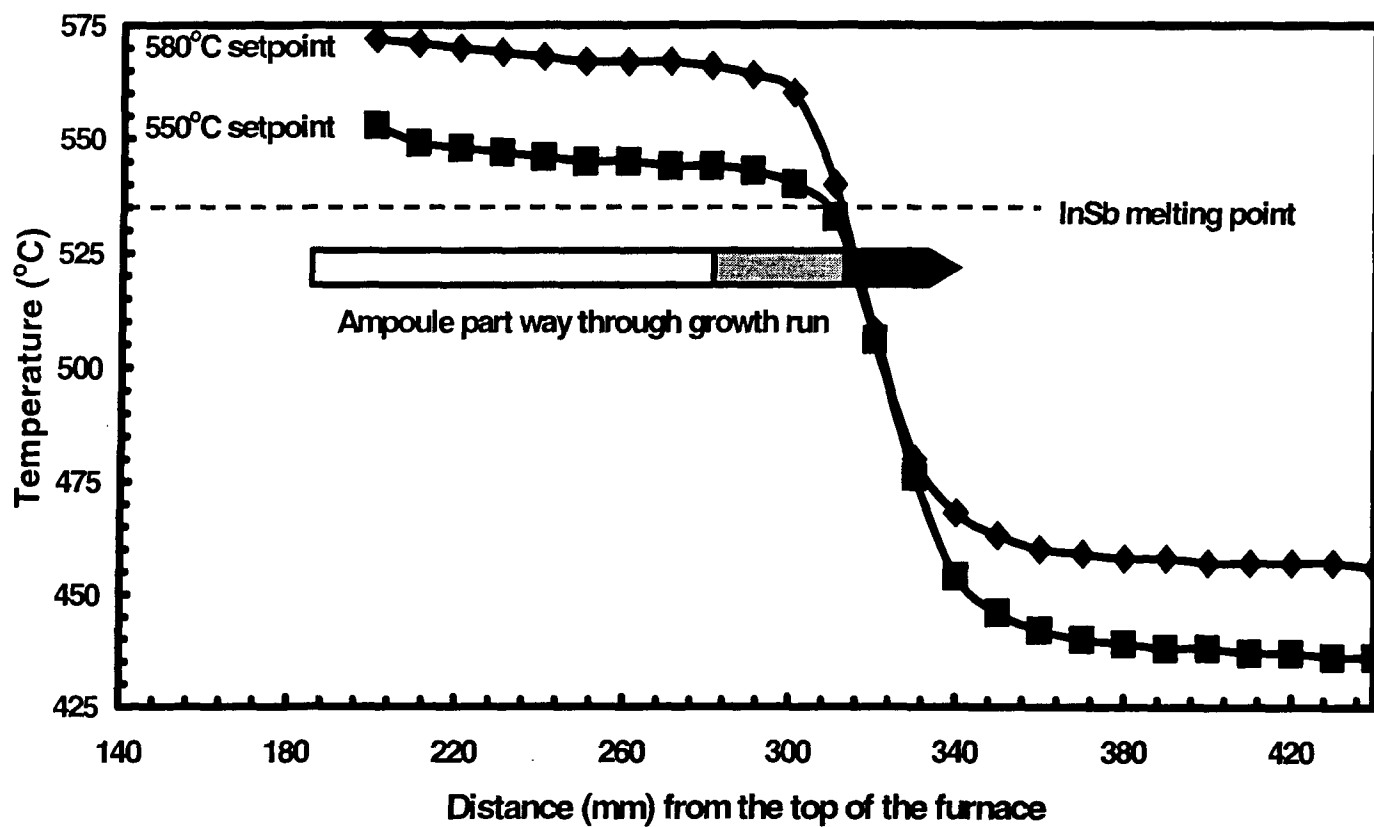


Fig. 1. Furnace temperature profiles for two different set points and the ampoule position in the furnace part way through an experiment.

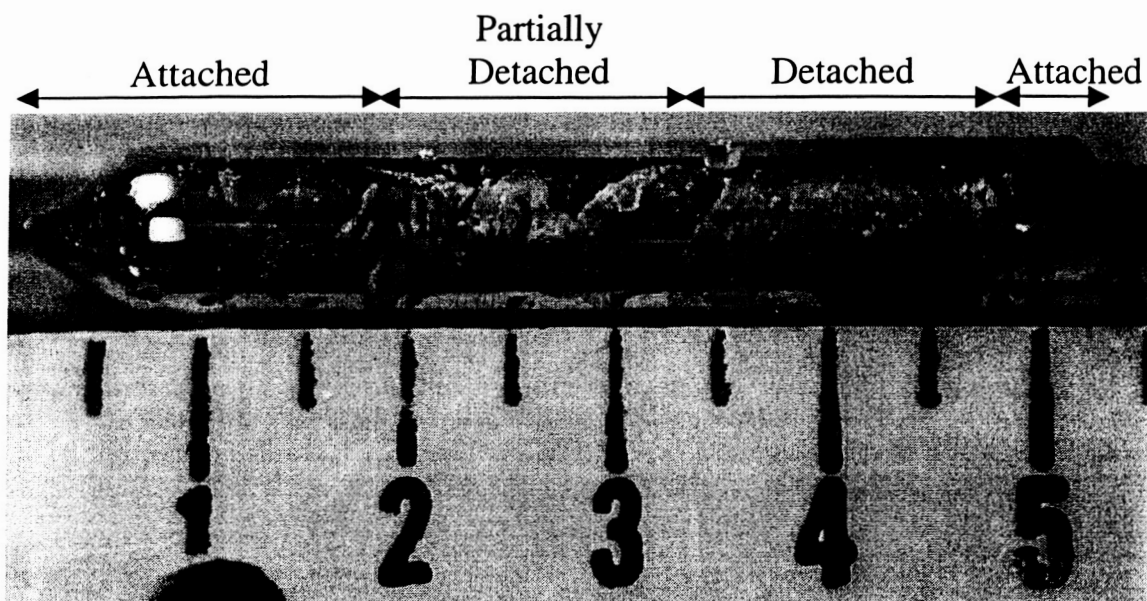


Fig. 2. Photograph of ingot 6, frozen at 10 mm/h in a BN-coated ampoule that had been backfilled with 20 kPa of forming gas prior to sealing. Scale is cm.

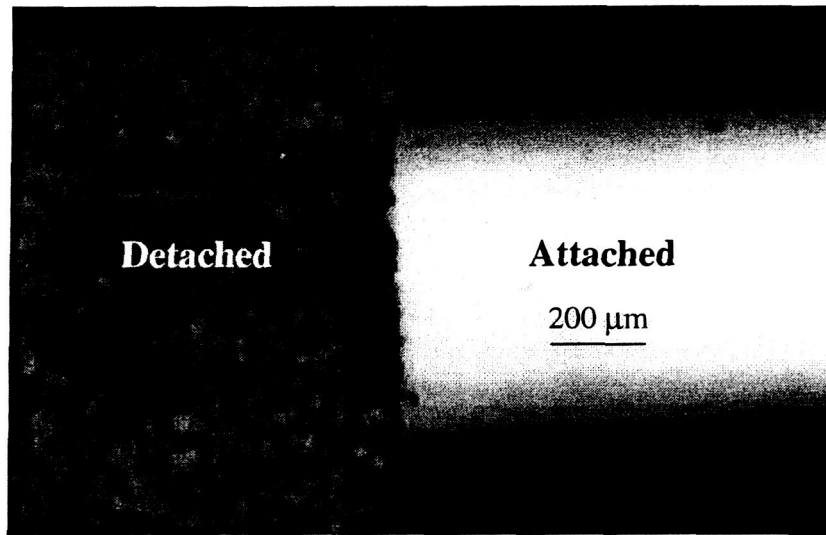


Fig. 3. Photomicrograph of a portion of the surface of ingot 6 shown in Fig. 2, taken at 50 X.

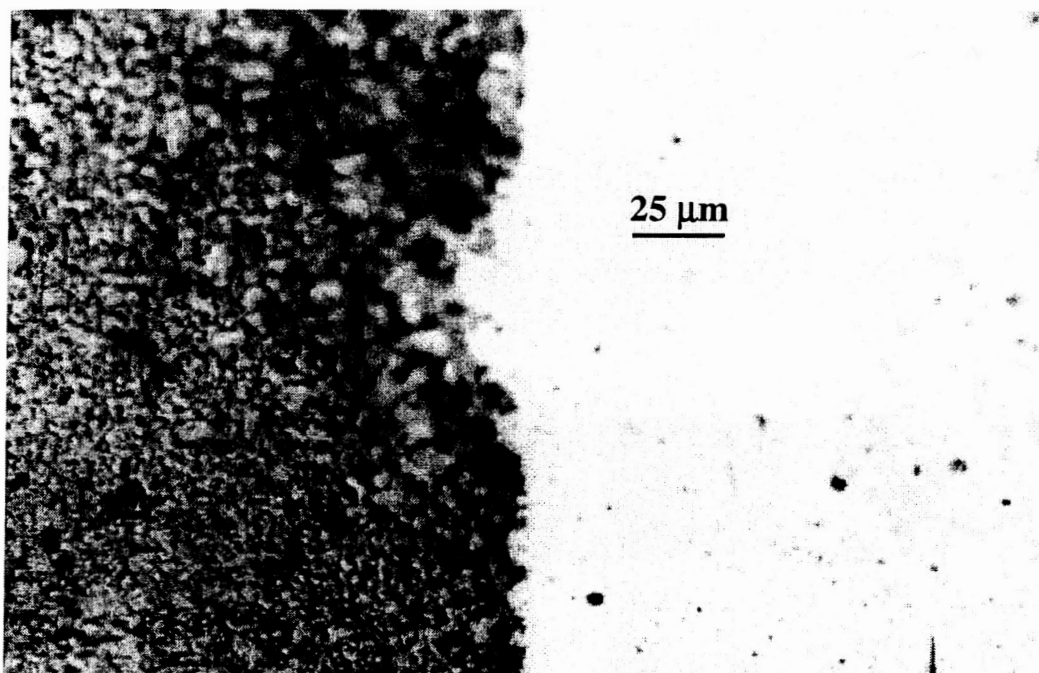


Fig. 4. Close-up of boundary between detached and detached sections of ingot 6, shown in Figure 3. Taken at 400 X.



Fig. 5. Photograph of Ingot 19 showing attached regions with small bubble voids and large cavities.

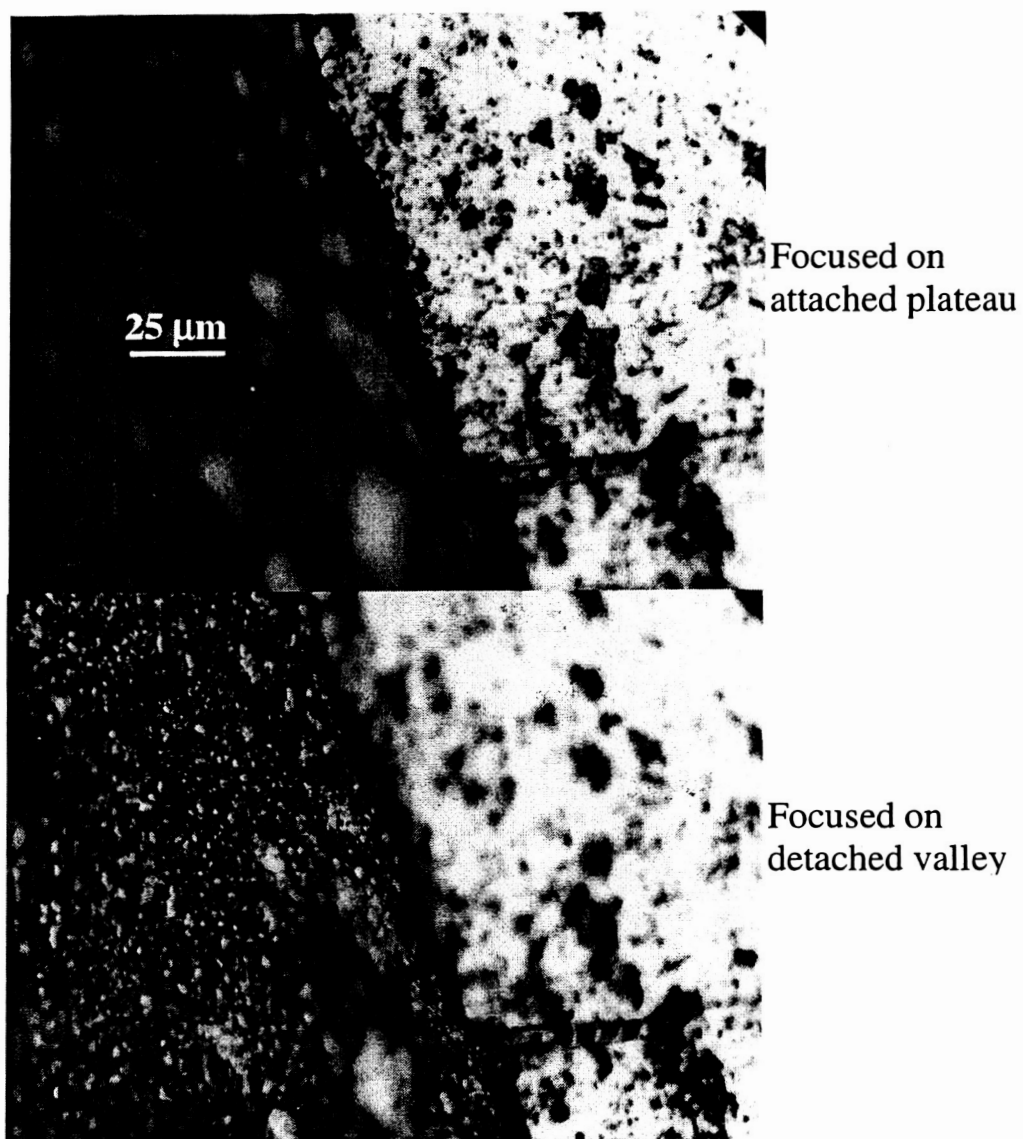


Fig. 6. Boundary between attached and detached sections of ingot 15. Taken at 400 X.

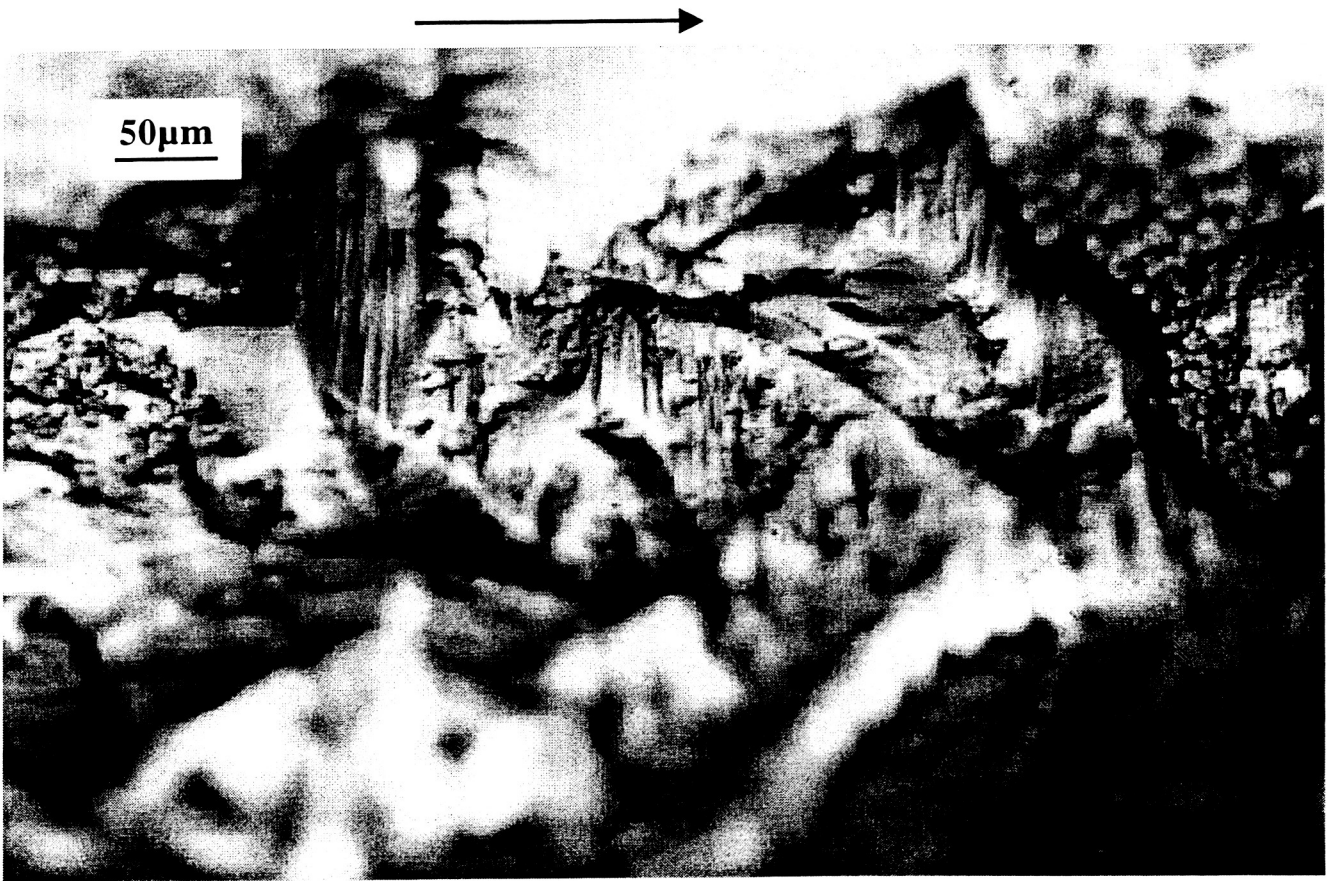


Fig. 7. Microfacets and steps on a detached section of Ingot 18 at 200 X.  
The arrow indicates the growth direction.



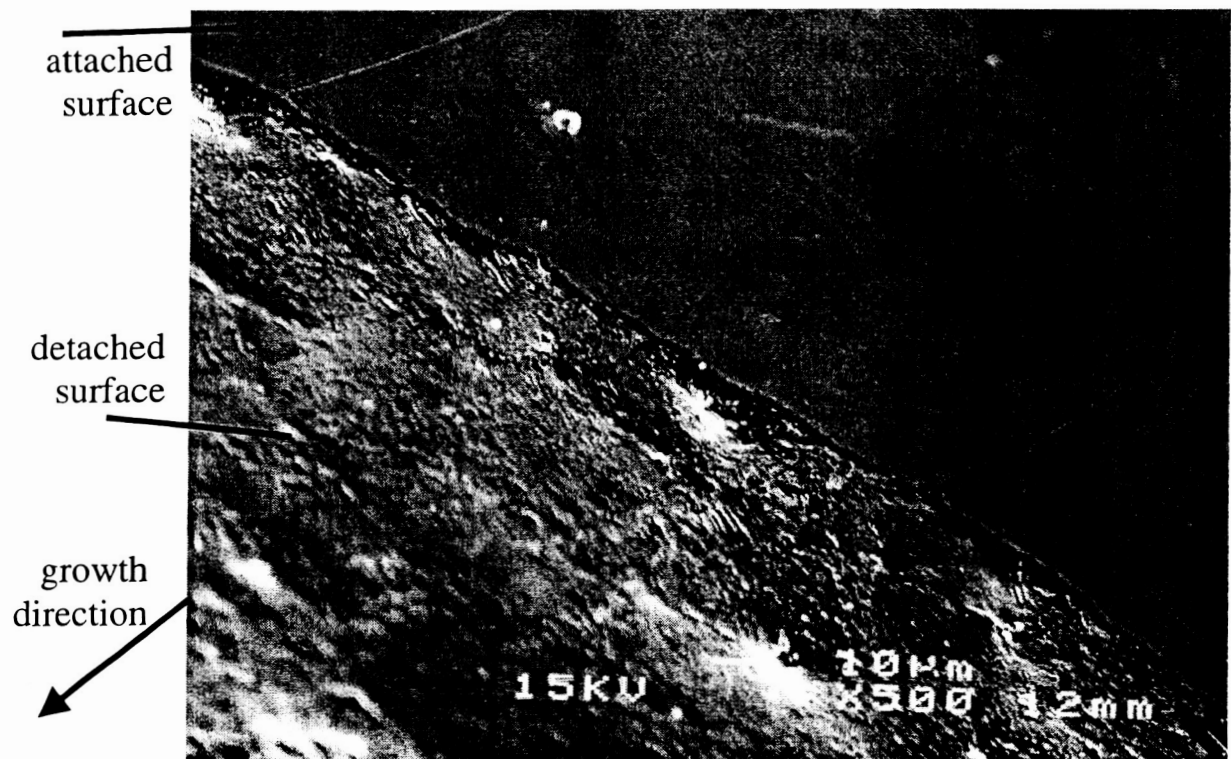


Fig. 8. Scanning electron micrograph of Ingot 6, rotated 60° and tilted 60° at 500X.

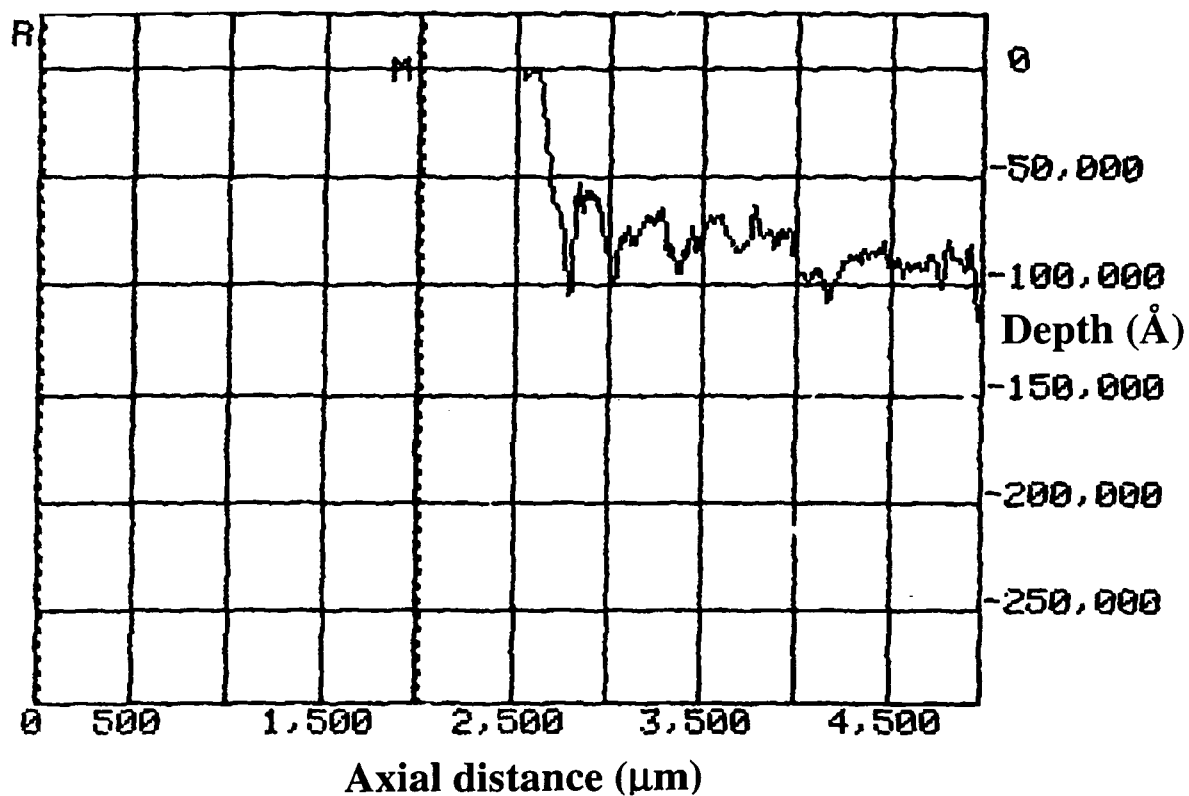


Fig. 9. Topography of the transition from attachment to detachment on ingot 6 via profilometer scan lengthwise along the surface. The attached portion is on the left and the detached on the right.

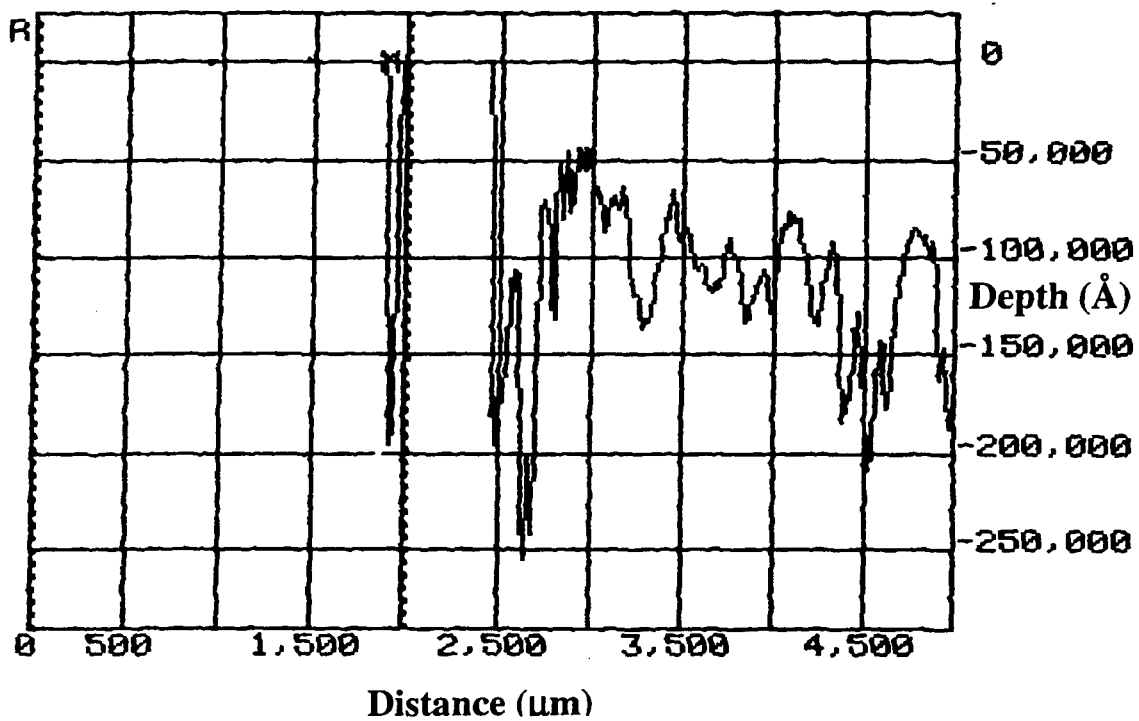


Fig. 10. Topography of the transition from attachment to detachment on ingot 15 via a profilometer scan lengthwise along the surface. The attached portion is on the left and the detached on the right. Note the gas bubble in the attached portion.